Hungarian Agricultural Engineering

N^{O} 25/2013

PERIODICAL OF THE COMMITTEE OF AGRICULTURAL AND BIOSYSTEMS ENGINEERING OF THE HUNGARIAN ACADEMY OF SCIENCES

Published by

Szent István University, Gödöllő
Faculty of Mechanical Engineering
H-2103 Gödöllő, Páter K. u. 1.
Dean: Dr. István SZABÓ

and the
ORGANISING COMMITTEE
of

SYNERGY
&
TECHNICAL DEVELOPMENT

Gödöllő, December, 2013

EXPERIENCES WITH MICROWAVE PRE-TREATMENTS OF SWEET WHEY PRIOR TO MESOPHILIC ANAEROBIC DIGESTION

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Abstract

In our work we focused on the examination of the applicability of microwave pre-treatments of sweet whey prior to anaerobic digestion. To quantify the structural change of organic matters of sweet whey biochemical oxygen demand (BOD), chemical oxygen demand (COD) and biogas yield were used as control parameters. Our results show that microwave pre-treatments are suitable to enhance the biodegradability of sweet whey. It was proved that the flow rate and ate specific power intensity has also effect on the biodegradability and biogas production of whey. Increased intensity and decreased flow rate has led to decreased solubility of organic matters of whey.

Keywords

microwave, sweet whey, biodegradability, anaerobic digestion

Introduction

The world is facing an energy crisis due to increasing concern related to fossil use, i.e. environmental impact, climate change, finite availability and security of supply. Rapidly rising motor fuel prices and the enhanced needs for them has stressed to increase the biofuel production. Biofuels are liquid or gaseous fuels made from plants and residues such as agricultural crops, municipal wastes and agricultural or forestry by-products.

Professionals in food industry companies face the high disposal costs of bio-wastes and the rising price of energy sources. Nowadays the renewable energy generation can be often connected to waste management technologies. For example since an effective utilization of food industrial biomass waste has desired, the establishment and optimization of an efficient biogas production process from these waste materials is very important from perspectives of both energy and environmental issues.

Food industry generates a huge amount of liquid and solid organic waste and by-products. Beside the considerable environmental risk of waste, it has a good potential to indirect bio-energy production for example in anaerobic digestion (AD) process. Biofuel production from agri-food wastes can also contribute to make waste management more socially acceptable, sustainable and cost effective (Nagy and Farkas, 2013). The anaerobic fermentation is a complex biological process developed in the absence of oxygen and in the presence of methanogenic bacteria, that transforms the organic substance into biogas (or biological gaseous mix), composed mainly from methane and carbon-dioxide (Kalmár et al., 2010).

Digestion is the oldest technology for waste stabilization and however less final waste sludge production can be achieved by controlled anaerobic decomposition. It is verified, that the biological degradability of organic matter of processed raw materials – such as solid wastes, sludge, lignocelluloses contained by-products- has effect on the rate of digestion. Because of non-biodegradable components and large molecules (proteins, polysaccharides) of raw materials different kind of pre-treatments are required to achieve an appropriate and economic ethanol and biogas yield.

Whey is an important by-product of the dairy industry, in the case of conventional cheese technology the final volume of whey is about 85-90% of the volume of processed milk. Two main whey types are produces in dairy technologies, acid whey and sweet whey (or cheese whey) depend on the procedure of casein precipitation. The principal components of whey are lactose, proteins and mineral salts. Approximately 150 million tons of whey disposed in the environment world-wide every year mainly in developing region (Leite et al., 2000, Saddoud et al., 2007). It represents a large-scale loss of resources and causes a strong environmental load because of the high organic matter content of whey and whey contained dairy wastewater.

The conventional waste treatment process is itself not suitable for producing stabilized whey waste for direct disposal (Siso, 1996). The technology of ethanol fermentation from whey is developed in several countries. For instance several distillers producing ethanol from whey are in commercial operation in Ireland, the USA and New Zealand, where about 50% of cheese whey is used to ethanol production (Mawson, 1994; Siso, 1996). In most cases, the bio-ethanol producing from non-concentrated whey can be unprofitable, because of the low ethanol concentration in fermentation broth the distillation process demands a lot of energy and it is uneconomical.

In Hungary the utilization of whey and membrane separated fractions of acid whey is used in whey based food industrial product. The whey is also could be appropriate as raw material for anaerobic digestion or animal feeding. Whey can be characterized by high chemical oxygen demand (COD) and biochemical oxygen demand (BOD) and more than 90% of 5 days BOD is caused by lactose content (Kisaalita et al., 1990). In the case of cheese whey the average fat content is less than in the acid whey and therefore the specific biogas and methane product is less.

Despite the many theoretical advantages, the anaerobic digestion is not widespread in the practice of dairy industry due to low dry matter content of whey, rapid acidification and the problems of slow reaction, which causes a longer hydraulic retention time in a continuous bio-system (Malaspina et al., 1996). Whey digestion process usually is carried out two major stage, the first involves the conversion of complex compounds to simple materials (for instance lactose into volatile acids, or polymers into monomers), in the second stage the end-products of fermentation process are transformed into mainly methane and carbon-dioxide by methanogenic bacteria (Göblös et al., 2008; Cohen et al., 1994).

Some pre-treatments assist or accelerate the hydrolysis of macromolecules or enhanced volatilization. The most commonly used process is the mechanical and combined (thermal and acidic or alkalic) methods as pre-treatments of biogas and bio-ethanol technologies, but there are some experimental lab-scale and pilot scale intensive system (assisted by microwave and ultrasound) to rapid digestion. Among pre-treatments, microwave irradiation alone or combined it with other thermal and chemical methods is considered as an intensive process with short process time and good ability to accelerate the hydrolysis stage of AD process and to enhance the specific biogas production.

The major advantage of MW heating over conventional thermal methods is the volumetric heating, which leads to faster heat and mass transfer and shorter process time. Application of MW irradiation combining with the oxidation process, such as ozonation, can also be considered to be promising technology as pre-treatment before AD of high organic matter containing but less degradable sludge (Beszédes et al., 2009). Energy transfer carried by microwave irradiation affect the biodegradability of materials in two ways. Thermal effect is expressed in the increase of internal pressure of intracellular liquor caused by internal heating and rapid evaporation, which altogether can lead to cell wall disruption (Géczi et al., 2013). The non-thermal effect of high frequency electromagnetic field contributes to alter the structure of macromolecules with polarization of side chains and breaking of hydrogen bounds (Park et al., 2004; Lakatos et al., 2005).High efficiency of MW treatments in the biomaterial processing and also on the rate of chemical reactions is often explained by the non-thermal effects of microwaves due to the direct interaction of electromagnetic field with molecules (Leonelli and Mason, 2010). MW irradiation has been successfully adopted as pre-treatment method via the high energy dissipation of polar compounds of sludge.

There are several studies concluding that the MW method has advantages over the pre-treatment process operating by conventional heating (Toreci et al., 2009). Increasing effect of MW irradiation on anaerobic digestion of organic solid waste was found, considering the substrate to bring 78% improvement was achieved after MW pre-treatment (Shahriari et al., 2012). Zheng et al. (2009) reported that microwave heating of primary municipal sludge resulted in 2.5 fold increases in soluble organic matter content related to the control at a pre-treatment temperature of 90 °C.

Thermal and a-thermal effects of the microwave (MW) irradiation play role in the "hot-spot" overheating phenomena, and the different dielectric parameter of cell components led to selective heating manifested in the different thermal stress, which contributes in the intensive degradation of cell wall components such as cellulose and pectin (Banik et al., 2003). MW pre-treatment solely has verified positive effects on cell wall destruction and releasing of organic matter into the soluble phase, but combining of it with addition of chemicals such as alkali, acid and oxidizer agents cause synergetic mechanism to accelerate the decomposition under aerobic and anaerobic condition., as well (Cheng et al., 2011).

Materials and methods

Sweet whey was used for the measurements, which is originated from a dairy works (Sole-Mizo Ltd., Szeged, Hungary). For the microwave (MW) pre-treatments concentrated whey fractions obtained from membrane separation process were also used to test the efficiency of MW process on whey with higher organic matter content. The membrane separation was carried out by 10 kDa ultrafiltration (UF) membrane made from polyethersulfone (PES). Components of samples were analyzed by Bentley 150 type infrared photometric milk analyzator. Main characteristic of processed samples are given in Table 1.

Table 1. Main characteristic of whey and concentrated whey

	Protein [w%]	Lactose [w%]	Fat [w%]	Total solids [w%]
Whey	0.47±0.13	2.61±0.04	0.18±0.01	3.24±0.07
Concentrated whey	0.73±0.16	3.59±0.09	0.34±0.08	5.36±0.24

MW pre-treatments were performed in a tailor-made microwave system; containing a continuously irradiating magnetron with changeable power in the range of 110 W to 700 W operating at a frequency of 2450 MHz. Power of the continuously irradiating microwave magnetron is adjustable by varying of anode voltage through a transformer with variable voltage. Experiments were carried out in continuously flow system; volumetric flow rate was varied by the speed of peristaltic pump.

The chemical oxygen demand of sample was measured triplicated using colorimetric standard method (APHA, 2005). COD in supernatant was determined after separation by centrifugation (12,000 rpm for 10 minutes) and prefiltration (0.45 μm Millipore disc filter). The biochemical oxygen demand (BOD5) measurements were carried out in a respirometric BOD meter (BOD Oxidirect, Lovibond, Germany), at 20 °C for 5 days. Total organic carbon (TOC) content of sample was analyzed by Teledyn Tekmar Apollo 9000 type TOC analyzer using 750 °C furnace temperature.

Batch mesophilic biogas production tests were carried out triplicate in continuously stirred reactors equipped by Oxitop-C measuring head applied pressure operating mode (WTW GmbH, Germany). Bottles had a total volume of 500 mL with a free headspace of 350 mL for gas production. For each anaerobic digestion (AD) tests fresh mesophilic anaerobic seed sludge was used supplied from an anaerobic digester of the local municipal wastewater treatment plant (Szeged, Hungary). pH of the mixed sludge was adjusted to 7.6. Reactors were purged with nitrogen gas to remove oxygen from the bottles. AD reactors were placed in an incubator at 37±0.2°C in dark. Biogas volume produced in AD tests was calculated from the pressure increment of head space of sealed bottles

Results and discussion

In our experiments the effect of volumetric flow rate, microwave power intensity and the number of treatment were examined on the chemical oxygen demand (COD), biochemical oxygen demand (BOD) and the ratio of BOD to COD parameters. The BOD/COD shows the percentage ratio of biodegradable part of total organic matter content of processed whey.

Our results shown, that the concentrate fraction of membrane separated whey had lower biodegradability than the non separated whey; although the COD has increased during the membrane filtration. These results can be explained by the relative higher concentration of proteins and other macromolecules in the concentrate phase which enhance the total organic matter content and therefore the oxygen demand of chemical digestion but these types of components era known as heavier degradable by microorganisms.

The experimental results have verified that the microwave pre treatments are suitable to enhance the biodegradability of organic matter contents of whey what manifested in higher BOD_5 and increased BOD_5/COD values. Microwave irradiation has effect on the structure of macromolecules of whey therefore the solubility and the ability of microbial decomposition was enhanced, as well. Similar tendency in BOD and BOD_5/COD values have been experience after the microwave treatments of the concentrated whey.

Table 2. BOD_5 and COD of microwaved samples

Sample	qv [Lh^{-1}]	MW power [W]	Number of treatment	COD [kgm^{-3}]	BOD_5 [kgm^{-3}]	BOD_5/COD [%]
Whey	-	-	-	89.0	24.8	27.87
Whey conc.	-	-	-	100.0	22.1	22.09
Whey	6	290	1	102.1	41.1	40.26
Whey conc.	25	290	1	92.4	34.1	36.92
Whey conc.	6	700	1	91.9	44.8	48.73
Whey	25	700	5	100.2	43.6	43.55
Whey conc.	6	290	5	88.3	44.1	49.94
Whey	25	290	5	99.3	39.0	39.28
Whey	6	700	1	103.8	50.1	48.27
Whey conc.	25	700	5	93.5	44.1	43.86
Whey	15.5	490	3	103.5	45.2	43.66
Whey	15.5	490	3	101.2	44.9	44.38
Whey	15.5	490	3	98.7	42.8	43.39

The most effective MW pre-treatment procedure produces 2-fold and 120% increment in biodegradability characterized by the BOD_5/COD parameter for whey and concentrated whey, respectively (Table 2).On the other hand, taking into consideration the increment of BOD_5/COD values, membrane separation as pre-treatment before MW operations was not beneficial. It was concluded, that the decreased volumetric flow rate due to the increased exposure time to MW irradiation resulted higher biodegradability by processing of whey and also concentrated whey. It was also experienced, that higher biodegradability was occurred after MW treatment with higher power intensity.

Beside the biodegradability obtained from aerobic degradation process the biodegradability in anaerobic bio-transformation process was also investigated. Efficiency of anaerobic digestion (AD) was characterized by the specific biogas yield, expressed in biogas production per total solid (TS) content of digested whey; and the generated biogas volume per total organic carbon (TOC) consumption during the AD process. Latter parameter gives information about the organic matter utilization during the biogas transformation and suitable to characterize the biodegradability under anaerobic condition.

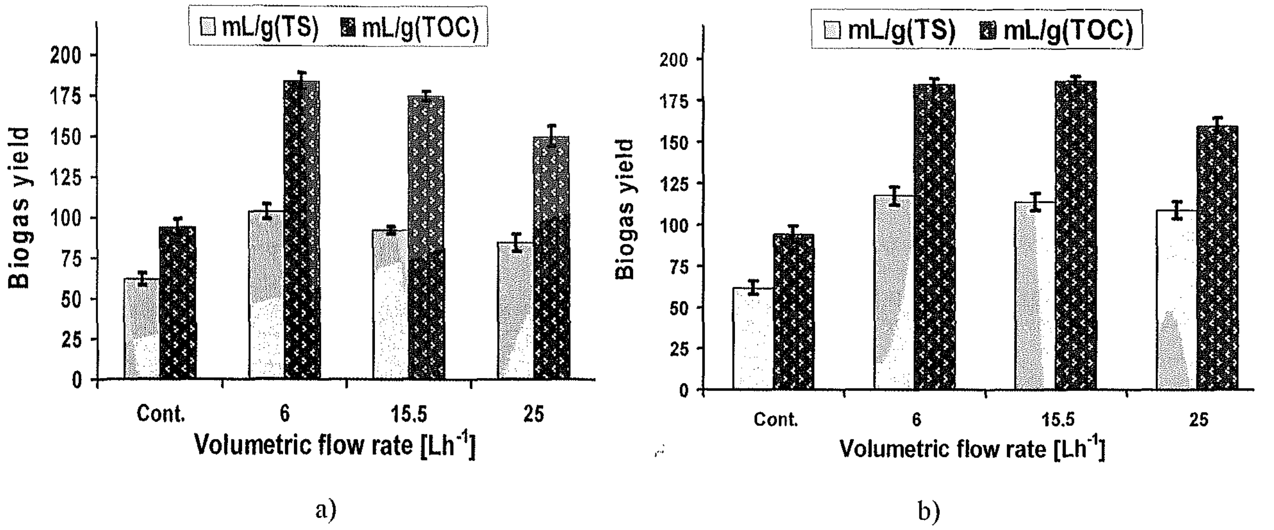

Figure 1.B Biogas yield of MW pre-treated whey samples by 280W (a) and 700W (b) MW power

Related to the control (non pre-treated) sample MW pre-treatments increased the biogas yield from whey. Specific biogas production of untreated sample (62 mL/gTS) could be enhanced to 100 mL/gTS applying 280W MW pre-treatment with flow rate of 6 L/h, and to above 120 mL/gTS after 700W microwave power with 6 and 15.5 L/h flow rates, respectively. Differences between the biogas yield obtained from MW pre-treated samples with different flow rates were higher using the lower – 280W- intensity than that of experienced after pre-treatment with the highest -700 W- power intensity.

Different tendency was observed between the effects of MW pre-treatment on values of the TS based and the TOC consumption based biogas yields, respectively (Fig. 1). There was not significant difference between the TS based biogas production of MW pre-treated samples applying different flow rates at MW power of 700W, but biogas production related to TOC consumption show that decreasing the flow rate (and therefore increasing the residence time in MW cavity resonator) had advantageous effect on the organic matter transformation to biogas, therefore the overall efficiency of AD process was higher.

Conclusion

In our work we focused on the investigation of MW pre-treatments on the aerobic and anaerobic biodegradability of whey co-digested by sewage sludge. Our results show that microwave pre-treatments are suitable to enhance the biodegradability of sweet whey due to the increased ratio of BOD to COD. It was proved that the flow rate and ate specific power intensity has also effect on the biodegradability and biogas production of whey. Increased intensity and decreased flow rate has led to decreased solubility of organic matters of whey.

Considering the specific biogas production was concluded, that MW pre-treatments increased the biogas yield of processed whey and membrane separated concentration of it by approximately 66%, and 42%, respectively. Data obtained from whey co-digestion with sewage sludge have revealed a 60% reduction in the lag-phase of AD process using high intensity MW pre-treatment (700W) with low (6 Lh-1) flow rate. Taking into consideration of the degree of anaerobic decomposition characterized by the ratio of produced biogas volume to the consumed organic matter (given as TOC) during the AD process can be concluded that the MW pre-treatments with optimized process parameters did not decrease the efficiency of the process.

Therefore, beside the sufficient organic matter removal efficiency, the biogas production could be increased with accelerated biotransformation what indicate a higher capacity for an industrial scale continuously fed digester

Acknowledgement

This research was realized in the frames of TÁMOP 4.2.4. A/2-11-1-2012-0001 „National Excellence Program – Elaborating and operating an inland student and researcher personal support system convergence program" The project was subsidized by the European Union and co-financed by the European Social Fund.

The authors are also thankful for the financial support provided by the Hungarian Scientific Research Fund (OTKA), under contract number K105021.

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